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14. ABSTRACT
The DOD HBCU/MI instrumentation award provided us a rare opportunity to acquire a Bruker Dimension FastScanTM Atomic Force Microscope (AFM) in 2015.The AFM instrument was installed in June 2015 and is greatly promoting our scientific research work and education programs. The Dimension FastScan allows scanning 10-20X faster than the traditional Icon scanner. Because of the scanning rates the AFM will permit us to study dynamic events such as melting, evaporation, crystallization, dissolution, self-assembly, membrane disruption,

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Report Title

Final Report: Acquire an Bruker Dimension FastScanTM Atomic Force Microscope (AFM) for Materials, Physical and Biological Science Research and Education

ABSTRACT

The DOD HBCU/MI instrumentation award provided us a rare opportunity to acquire a Bruker Dimension FastScanTM Atomic Force Microscope (AFM) in 2015.The AFM instrument was installed in June 2015 and is greatly promoting our scientific research work and education programs. The Dimension FastScan allows scanning 10-20X faster than the traditional Icon scanner. Because of the scanning rates the AFM will permit us to study dynamic events such as melting, evaporation, crystallization, dissolution, self-assembly, membrane disruption, sample movement tracking. To ensure that the state-of-the-art AFM is appropriately utilized by members of the Clark Atlanta University scientific community, two super users, Drs. Biswajit Sannigrahi and Guangchang Zhou were trained by the Senior Engineer for Product Service, Dr. Teddy Huang from the Bruker Nano Surface Division during the course of 4-day installation/training from June 8 to 11, 2015. They not only mastered the basic manipulation, troubleshooting and routine maintenance of the AFM instrument, but also were familiar with the other different modes of operation: mechanical, electrical, thermal, fluid imaging. The AFM is currently being utilized by several students to study the self-assembly of hairy nanoparticles and block copolymers. The Dimension FastScan® AFM as a new nanotool is boosting our research work and education programs.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received	Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received	Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received	Paper

TOTAL:

Number of Manuscripts:

Books

Received	Book

TOTAL:

Received Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
Ian Stubbs	0.00	
Reem Suliman	0.00	
Genefine Sapateh	0.00	
Amrit Sharma	0.00	
Azza Habel	0.00	
Hanif Uddin	0.00	
FTE Equivalent:	**0.00**	
Total Number:	**6**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
Biswajit Sannigrahi	0.00
Guangchang Zhou	0.00
FTE Equivalent:	**0.00**
Total Number:	**2**

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
ishrat Khan	0.00	
FTE Equivalent:	**0.00**	
Total Number:	**1**	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: 0.00

Names of Personnel receiving masters degrees

NAME
Total Number:

Names of personnel receiving PHDs

NAME
Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

See attachment

Technology Transfer

Annual Reports

For the DOD HBCU/MI Instrumentation Award Regarding

“Acquire an Bruker Dimension FastScan™ Atomic Force Microscope (AFM) for Materials, Physical and Biological Science Research and Education”

By

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Atomic force microscope (AFM) is an instrument capable of imaging and manipulating individual atoms and molecules, and plays a crucial role in the emergence of the field of nanoscience and technology. Currently, AFM has revolutionized the study of surfaces at the nanometer scale. AFM has proved its suitability in various applications, especially for chemical and biological applications. The technique has been adapted to work in various environments, for example, in liquid, at low temperatures, in high magnetic fields and so on.

Bruker is a global leading company in producing AFM instruments. The advanced model of high-speed atomic force microscopy (HS-AFM) is **Dimension FastScan® AFM.** It delivers extreme imaging speed with atomic resolution and Atomic PeakForce Capture™.

The very latest advances in atomic force microscopy techniques, including proprietary PeakForce Tapping® technology, enable researchers to discover new possibilities in mechanical, electrical and chemical applications. PeakForce Tapping has become the principal AFM mode with the fastest growing publication record. Applications range from biology to semiconductors, from data storage devices to polymers, and from integrated optics to measurement of forces between particles and surfaces. As the only major AFM manufacturer with a state-of-the-art probes nanofabrication facility and world-wide, application-specific customer support, Bruker is uniquely positioned to provide users the equipment, guidance, and support for all their nanoscale research needs.

The DOD HBCU/MI instrumentation award provided us a rare opportunity to acquire a dimension AFM with FastScan scanner shown in Figure 1. The AFM instrument was housed in our university last June and is greatly promoting our scientific research work and education programs.

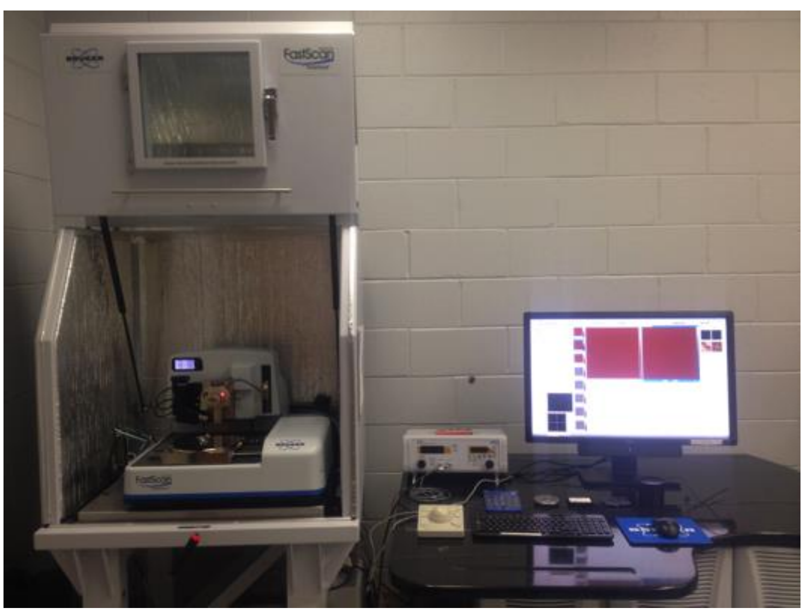


Figure 1. The AFM Instrument of Clark Atlanta University

Dating back to 2006, our university established the Center for Functional Nanoscale Materials (CFNM). The CFNM is the University's focal point for chemical, physical and biological science studies of nanometer-scale structures, their unique properties, and their integration into functional units. It fosters multidisciplinary research and education efforts involving faculty whose combined expertise spans the disciplines of a comprehensive research university, including the arts and sciences, and medicine, as well as other professional schools. The Center has been tremendous success in a time when number of doctorates awarded to African and Hispanic Americans are far less than their proportion in population. The Center has become a major base for fostering doctoral students from groups underrepresented in the science and technology areas. Currently, twenty-two graduate students are carrying out their research under the auspices of the CFNM. *The objectives of the state-of-the-art AFM are to advance research and education in nanoscience and nanotechnology and to achieve significant growth in research support.*

For this purpose, two super users, **Drs. Biswajit Sannigrahi** and **Guangchang Zhou** were trained by the Senior Engineer for Product Service, Dr. Teddy Huang from the Bruker Nano Surface Division during the course of 4-day installation/training from June 8 to 11, 2015. They not only mastered the basic manipulation, troubleshooting and routine maintenance of the AFM instrument, but also were familiar with the other different modes of operation: mechanical, electrical, thermal, fluid imaging. With the FastScan head, it also delivers imaging rates that increase throughput and general productivity as well as the new possibility of capturing dynamic events in movies comprising hundreds of captured images. These dynamic events could be melting, evaporation, crystallization, dissolution, self-assembly, membrane disruption, sample movement tracking etc. The Dimension FastScan allows scanning 10-20X faster than the traditional Icon scanner.

Peak Force Tapping is a proprietary Bruker scanning technique. The integration of TappingMode imaging techniques into a fluid environment makes this a powerful tool for studying fluid samples without artifacts caused by drying or excessive force. TappingMode is currently the most widely employed mode of AFM for fluid samples. With this technique, polymer latexes and biological samples can be directly imaged. PeakForce TUNA enables, for the first time, current imaging on extremely soft and delicate samples, as well as, superior tip lifetime for current imaging on hard samples. PeakForce TUNA also includes the quantitative nanomechanical property mapping suite of PeakForce QNM, thereby providing electrical information simultaneous with topography and mechanical property information (deformation, adhesion, DMT modulus, and dissipation).

Most AFM studies to date have been conducted at ambient temperature. This presents a substantial limitation when information about the thermal behavior of the sample surface is desirable. For example, performance of plastics is strongly temperature dependent due to their multiple phase transitions, such as melting, crystallization, recrystallization, as well as glass and sub-glass transitions. The Dimension™ Heater and Cooler Accessories

enable AFM to be performed down to -35°C and at elevated temperatures up to 250°C, with precise control while stepping or cycling through thermal ranges.

During the installation/training, Dr. Huang demonstrated how to image a liquid sample, determine nano-mechanical properties such as Yong's Modulus of samples, as well as measure surface potential of samples using PeakForce TUNA.

There are a huge number of improvements on the latest generation instrument including lower noisefloor and drift, huge software improvements, innovative new modes, a FastScanning head, new hardware accessories, a powerful controller with advanced features etc. The Dimension AFM with FastScan offers the following advantages.

- Flexibility of sample size
- Easy customization
- Motorized and automated controls/ ease of use
- Inherent stability and robust design
- Huge accessory array for multiuser experiment flexibility

To meet our research and education needs, the two super users have trained the following graduate and undergraduate students to use the AFM instrument.

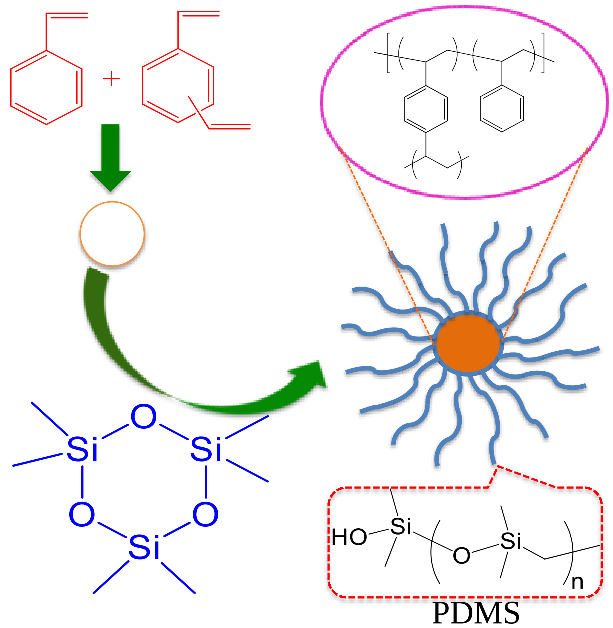
Reem Suliman (graduate student) **Genefine Sapatech (Graduate Student)**

Ian Stubbs (Graduate student) **Hanif Uddin (Graduate Student)**

Amrit P. Sharma (Graduate Student) **Azza Habel (Graduate Student)**

Tyquavious Kelley (High School Student)

Most importantly, the AFM instrument has been employed to investigate self-assembly of hairy nanoparticles synthesized by living anionic polymerization techniques (Figure 2). We synthesized a series of hairy nanoparticles with different content of polydimethylsiloxane (PDMS) (Figure 2).



Sample Code	Molar Percentage of PDMS in Hairy Nanoparticles (%)
ALW-002	32.52
ALW-004	51.99
ALW-005	73.36
ALW-006	54.40
ALW-008	56.17

!

Figure 2. Schematic diagram of synthesis and structure of hairy nanoparticles as well as their structural compositions shown in the right panel.

All hairy nanoparticle sample solutions in THF with the same concentration of 1.0 mg/mL were prepared and drop-casted onto the silicon wafers and allowed to dry in air. All hairy nanoparticle samples in their dried state were directly imaged by the AFM instruments. Figure 3 showed the AFM images of all samples with different content of PDMS. All samples indicated multiple-sized spherical morphology with different diameter ranges. Remarkably, the hairy nanoparticle sample (ALW-005) with 73% mol. of PDMS can form large spherical particles with different shapes because the small particles were able to connect and fuse together during evaporation of THF. To avoid fusing of particles and observe individual particles, diethyl ether (Et_2O) was used instead of THF. Similarly, four samples were imaged by the AFM instrument (Figure 4). Samples ALW-002 and ALW-004 showed particle clusters formed by their individual particles with diameter ranges of 153 ~ 176 nm and 355 ~ 441 nm respectively. Samples ALW-005 and ALW-008 showed almost uniform particles with different shapes. Interestingly, four individual particles connected together to form a symmetrical cluster with a size range of 1610 ~ 2030 nm (Figure 4D). The individual particles for each sample can be successfully observed using the low boiling point solvent, diethyl ether, due to its rapid evaporation avoiding fusing among particles. Therefore, we investigated the effect of solvent on the size and shape of particles. Four different solvent systems, namely, Et_2O, THF, Acetone and THF-Et_2O mixture, were used to prepare a series of

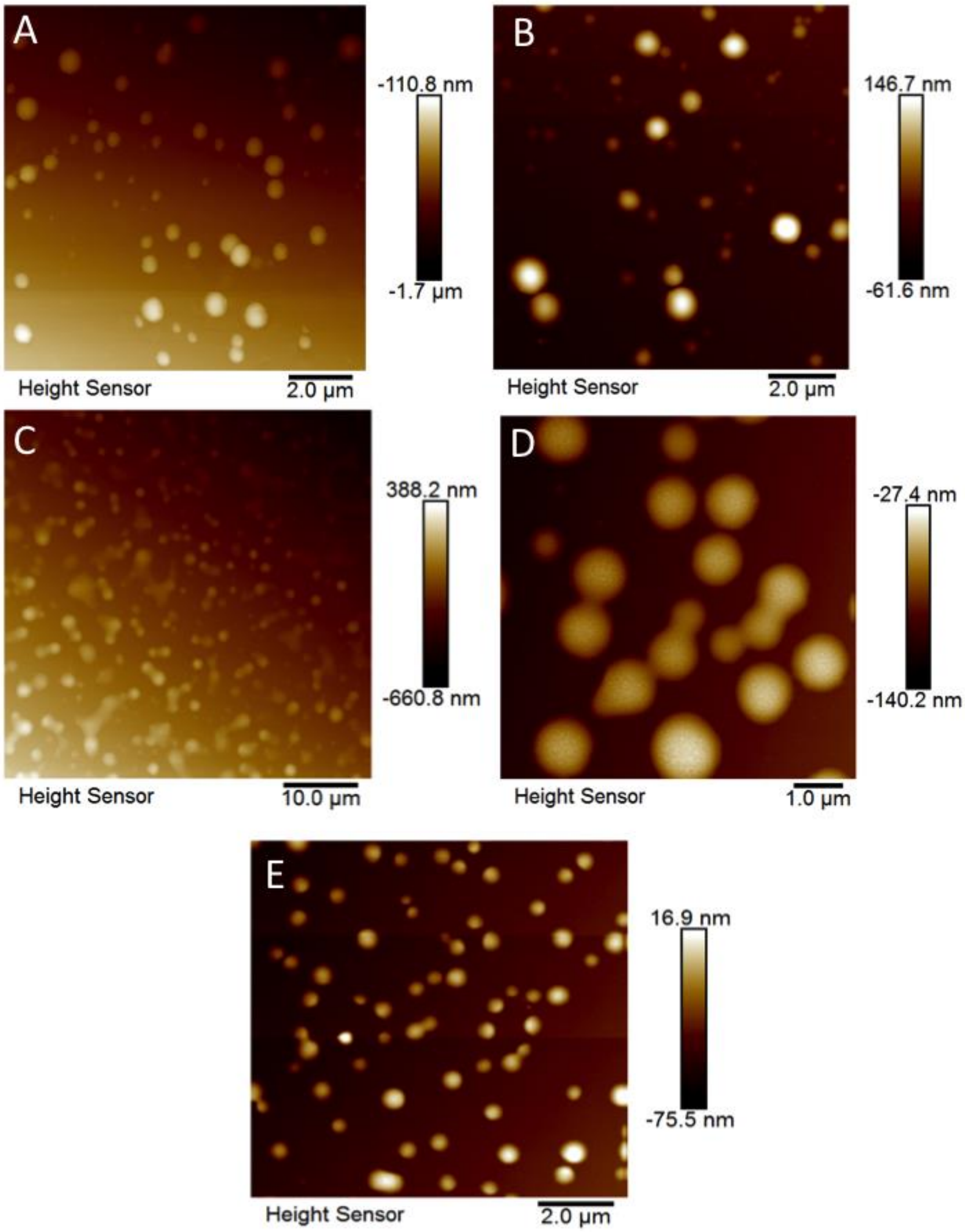


Figure 3. AFM images of the samples prepared by drop-casting THF solutions of hairy nanoparticles and air-dried (Sample concentration: 1.0 mg/mL). (A) ALW-002 (particle size range: 416 ~ 791 nm in diameter). (B) ALW-004 (particle size range: 460 ~ 960 nm in diameter). (C) ALW-005 (particle size range: 1280 ~ 2080 nm in diameter). (D) ALW-006 (particle size range: 673 ~ 1510 nm in diameter). (E) ALW-008 (particle size range: 460 ~ 660 nm in diameter).

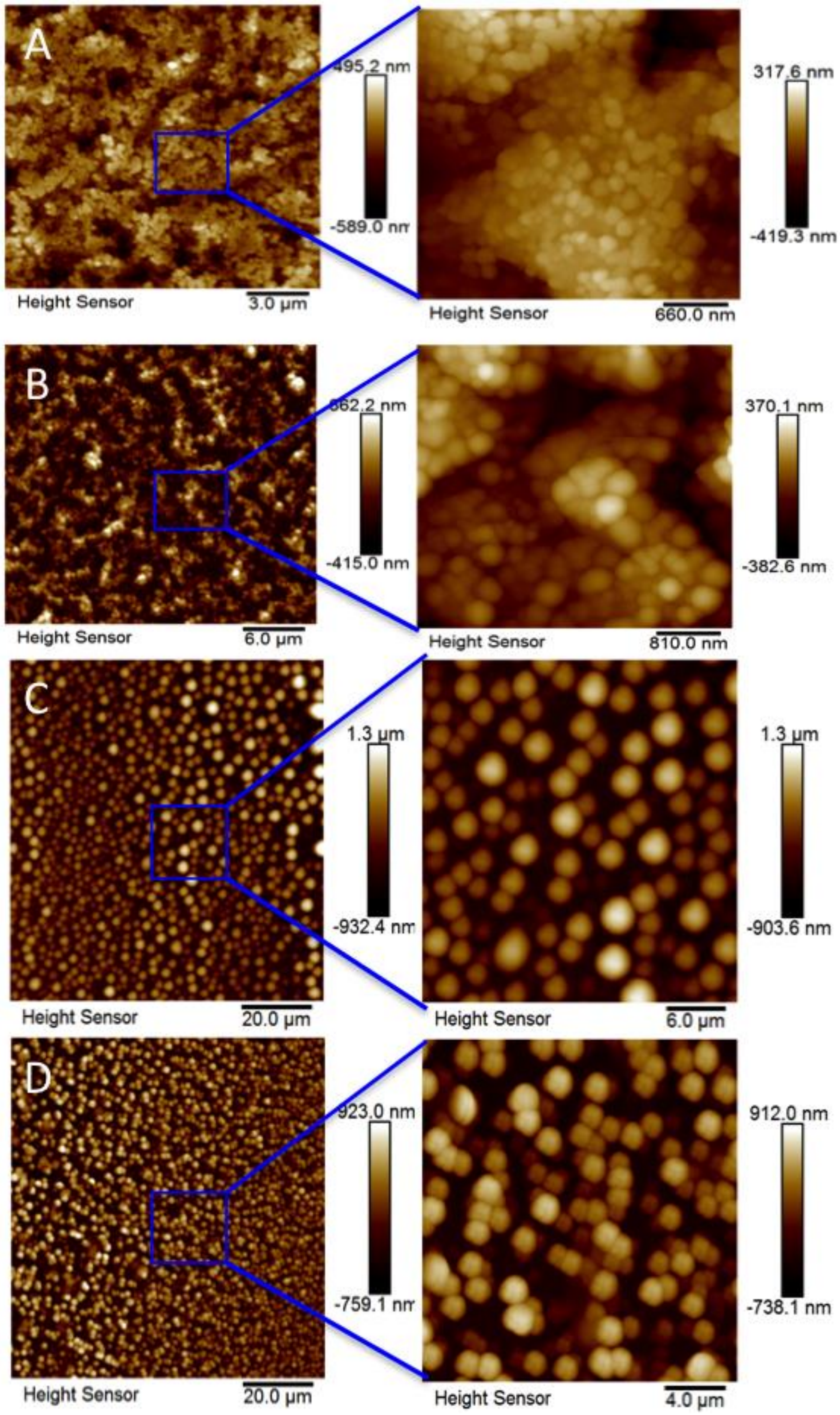


Figure 4. AFM images of the samples prepared by drop-casting Et_2O solution of hairy nanoparticles and air-dried (sample concentration: 1.0 mg/mL). (A) ALW-002 (particle size range: 153 ~ 176 nm in diameter). (B) ALW-004 (particle size range: 355 ~ 441 nm in diameter). (C) ALW-005 (particle size range: 2260 ~ 4140 nm in diameter). (D) ALW-008 (particle size range: 1610 ~ 2030 nm in diameter).

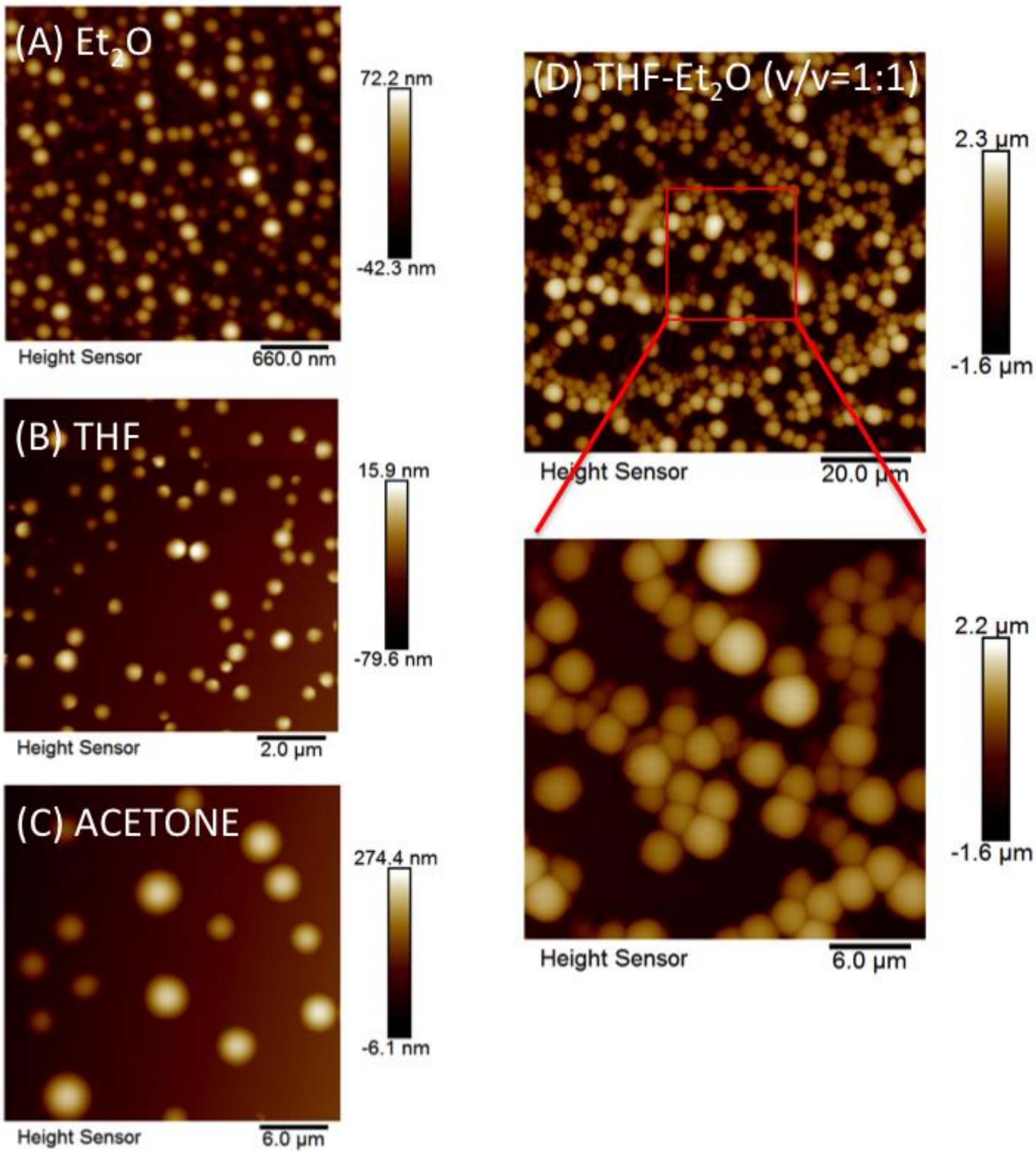


Figure 5. AFM images of the samples of the hairy nanoparticle ALW-008 prepared by drop-casting solutions of different solvents (sample concentration: 1mg/mL). Different solvent solution can lead to different particle sizes. The particle size ranges are 145 ~ 230 nm for Et_2O (A), 406 ~ 719 nm for THF (B), 2500 ~ 4240 nm for Acetone (C) and 2190 ~ 4280 nm for THF-Et_2O mixture solvent (v/v: 1:1) (D).

sample solutions with the same concentrations of 1 mg/mL for the representative sample, ALW-008. The prepared sample solutions were further drop-casted onto the silicon wafers and air-dried and followed by AFM imaging. Figure 5 showed the AFM images of the sample ALW-008 obtained using different solvent systems. The sample ALW-008 was able to form spherical particles in each solvent system. However, the particle size ranges are obviously different as indicated by the particle size ranges of 145 ~ 230 nm,

406 ~ 719 nm, 2500 ~ 4240 nm and 2190 ~ 4280 nm for Et_2O, THF, acetone and THF-Et_2O mixture solvent (v/v: 1:1) respectively. The sample ALW-008 can form larger particles with multiple-sizes in both THF and acetone because of particle growth during the solvent evaporation. It is very promising that the sample can form almost uniform particles in either the single Et_2O solvent system or Et_2O-containing mixture solvent (Figure 5A and 5D).

Unlike drop-casting, spin coating enables to quickly and easily produce very uniform films from a few nanometres to a few microns in thickness. Two different sample solutions in Et_2O was spin-coated onto the silicon wafers, and further imaged by the AFM instrument (Figure 6). The sample ALW-002 with 32.5% mol. of PDMS can form rope string-like organized suprastructures. The sample ALW-002 with 56% mol. of PDMS can form uniform equilateral triangle clusters with all sides the same length of 738 nm. So, spin-coating technique can help produce very ordered suprastructures.

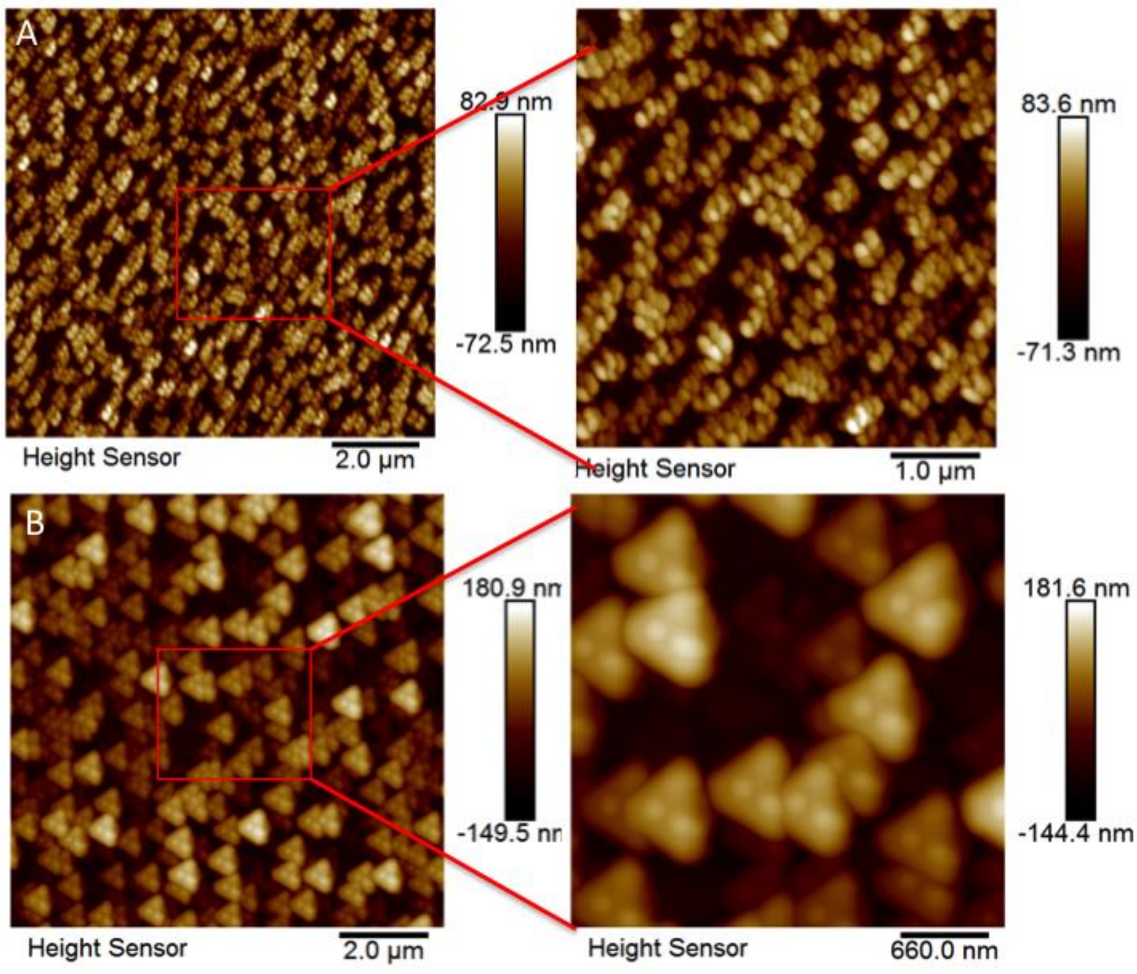


Figure 6. AFM images of the samples prepared by spin-coating Et_2O solution of hairy nanoparticles and air-dried (Sample concentration: 1.0 mg/mL). (A) ALW-002; (B) ALW-008.

In addition, we also imaged the other nanostructured materials. For example, we unzipped the commercially available multi-walled carbon nanotubes (MWCNT) (D L: 110 ~ 170 nm 5 ~9 μm) and further imaged the unzipped MWCNT-nanoribbon products using AFM (Figure 7A). The produced MWCNT-nanoribbons have quite rational dimensions (W L: 399 nm 2.65 ~ 4.38 μm) based on the calculation of circumference of MWCNT. This confirmed the successful unzipping of MWCNT. A single nanofiber with a variable diameter from 2.72 μm to 4.92 μm can be directly imaged using the AFM instrument (Figure 7B).

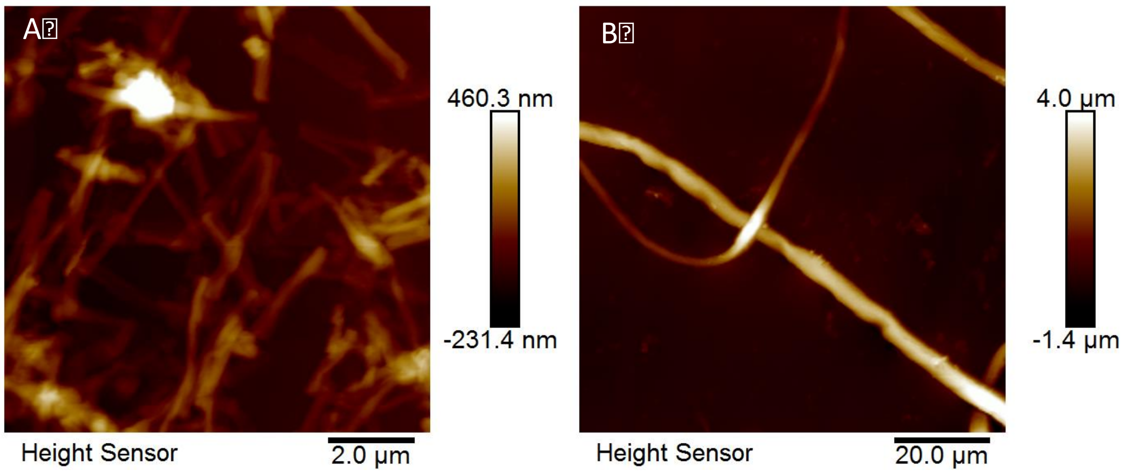


Figure 7. AFM images of unzipped multiwalled carbon nanotube (MWCNT)-nanoribbons (A) and nanofibers (B).

In summary, the Clark Atlanta University (CAU), a historically black university, is strongly committed to enhancing its research and education capabilities in science and engineering. A new 200,000 square foot Research Center for Science, Engineering and Technology, currently fosters interdisciplinary research programs and trains minority scientists. The CFNM center is equipped with many state-of-the-art instruments to support a lot of federal research projects. However, the Dimension FastScan® AFM as a new nanotool is boosting our research work and education programs. Currently, it is open access on a routine basis to all faculty, staff and students.